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Performance analysis of the cold flow model of a second generation chemical looping combustion reactor system

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Abstract

A 150kW$_{th}$ second generation chemical looping combustion reactor system has been designed. It is a double loop circulating fluidized bed meant to achieve high solids circulation and be flexible in operation. Attention was also focused on the usage of industrial solutions and compactness, to go towards commercialization and pressurization as a further step. Both its hydrodynamic behaviour and design solutions were investigated by means of a full scale cold flow model. First the design of the nozzles and the share of kinetic losses were verified, together with the solids flow/flux measurements reliability. The air reactor and fuel reactor were then tested separately monitoring their entrainment capabilities and pressure/particles distribution, with main focus on finding the best way of operating the loop-seals and cooling panel configuration. The overall reactor system (combining air and fuel reactor) was tested achieving results close to the design values. Finally, some solutions to further improve its performance are proposed.

Keywords: Chemical Looping Combustion, Double Loop Circulating Fluidized Bed, Cold Flow Model, Industrial solution, Pressurization

1. Introduction

Within the CO_2 capture technologies the Chemical Looping Combustion (CLC) is one of the most promising both for costs and net efficiencies [1]. It is an unmixed combustion process with inherent CO_2 separation, commonly realized by means of two interconnected fluidized bed reactors. It takes place in two steps where a metal powder, working as a solid Oxygen Carrier (OC), gets cyclically oxidized and reduced carrying the oxygen from one reactor to the other. First the OC has a strong exothermic reaction with the oxygen of the fluidizing air in the Air Reactor (AR). Afterwards the oxidized OC is sent into the Fuel Reactor (FR) and its oxygen reacts with the fuel, endothermically or slightly exothermically, depending on the OC material and fuel used, generating an almost pure stream of CO_2 and steam.

SINTEF Energy Research and the Norwegian University of Science and Technology (NTNU) have designed a 150kW$_{th}$ atmospheric CLC reactor system. The chosen design solutions are aiming at high operational flexibility and fuel conversion as well as compactness for the prospective of pressurizing the reactor as a further step. It consists of

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two Circulating Fluidized Beds (CFB) interconnected by means of a bottom extraction/lift and divided Loop-Seals (LS) in a two loops architecture: Double Loop CFB (DLCFB) shown in Figure 1. The AR is meant to operate in fast fluidization regime while the FR both in turbulent and fast fluidization. The abovementioned loop-seals are designed as double loop-seals. The purpose is both to avoid the gas mixing between the two reactors and to lead the flow of solids entrained by one reactor into the other one or re-circulate it back to the reactor of origin. They are fluidized by means of three bubble cap nozzles: one below the downcomer (central), one just below the internal return leg (to lead the mass back to the reactor of origin) and one below the external return leg (to lead the entrained mass to the other reactor). In addition there will be lateral steam injections in the downcomers, just above the loop-seals (Figure 1). Because of the smaller amount of fluidizing gas available in the FR, compared to the AR, the bottom extraction will compensate the fact that the FR is not capable to entrain the same amount of solids as the AR. The system is cooled by means of lateral protruding Cooling Panels (CP) inserted into the AR body. The DLCFB design and the way it faces industrial and scale up issues, as the latest (second) generation of CLC reactors does, has already been described in a detailed way by Bischi et al. [2]. In order to achieve the aforementioned objectives, the hydrodynamics as well as many of the design solutions of such CLC reactor system, need to be qualitatively tested in a Cold Flow Model (CFM) without chemical reactions [3].

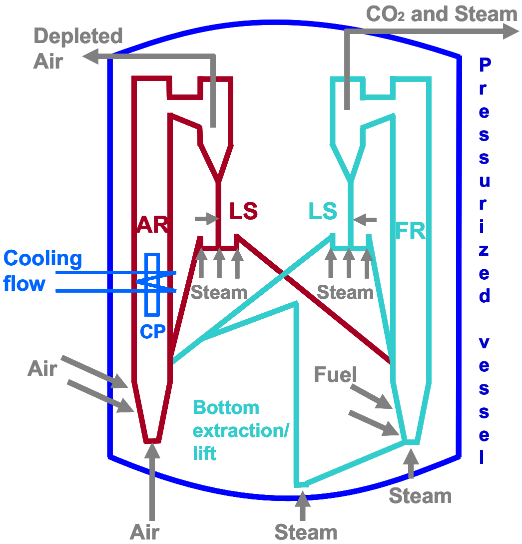

Figure 1. Process diagram of the Double Loop Circulating Fluidized Bed (DLCFB) reactor system concept [2].

2. Experimental set up and procedure

A polycarbonate CFM has been built and commissioned. It is a full scale (1:1) exact copy of the 150kW$_{th}$ hot rig design. In this way it was possible to reduce the wall effects to get more reliable design verification [4]. The two reactors are 5 meters high; the AR has a diameter of 0.23 m while the FR 0.144 m. In addition the powder characteristics as well as the operating conditions were chosen in order to end up in the same fluidization regime as the hot rig, i.e. fast CFB regime according to the empirical classification of Grace [5]. The selected material representing the oxygen carrier is a Ferro-Silicon alloy with a density of almost 7000 kg m^{-3} and a d_{50} of 34 micrometers and rounded irregular shape. These particles end up in the group A of the Geldart diagram and because of their high density are very close to the boundary with the group B [6]. An important share (above 20%) of the particle size distribution used in the tests has a diameter smaller than the foreseen critical one where the Van der Waals cohesion forces start to play a decisive role into fluidization properties [7, 8]. Anyhow, as long as the main interest for such fine particles is in the catalyst applications (thus lighter) it is quite rare to find information about high density Geldart A particles in open literature. The fluidizing gas is air and the nominal flows are selected to give a velocity of 2.2 m s^{-1} in the reactor bodies. Details of the scaling strategy can be found in Bischi et al. [9].

The rig is equipped with differential pressure transmitters distributed along the reactor bodies, cyclones and bottom of the loop-seals. At the reactor exit there is one common filter box with a frequency controlled fan so it is possible to obtain the desired backpressure, which will be the same for both the AR and the FR unless the valves at the cyclones exit are used to differentiate them. The filter box is also used to collect the powder losses and in this way monitor the cyclones efficiency and the inventory in the system. The solids entrainment is measured in two ways: a visual and an indirect way. The first one is relying on a visual measurement of the mass accumulation in the downcomer once the loop-seal fluidization is shut down. The indirect measurement is based on a perforated flap valve located in the downcomer. This way the gas coming from below fluidizes the amount of powder accumulating on the flap valve, when closed. If the minimum fluidization condition is reached, the entrained solids flux, thus flow, can be derived from the gradient of the pressure drop (ΔP) across the flap valve due to the mass accumulated [10]:

$$G_s = \frac{d\Delta P}{dt} \cdot \frac{1}{g} \cdot \frac{A_{downcomer}}{A_{riser}} \quad . \tag{1}$$

All the pressure measurements used to evaluate the reactors performance are an average value of ten minutes steady state operation. In addition the pressure in the more sensitive points (e.g. bottom loop-seal) was constantly monitored and experiments with too high pressure fluctuations (above 40 mbar) were labelled as unstable. The solids flows/fluxes were measured at least two times and an average value was presented. In case of a standard deviation of the measurements bigger than the 10% of the average a third measurement was taken.

3. Results and discussion

The CFM was first tested without particles to check the fluidizing nozzles design and the share of kinetic pressure losses. The pressure losses across the nozzles were measured as a function of the gas flow injected through the nozzles. In this way it was possible to evaluate if the ΔP of each nozzle is in the proper range: above 20% [11] of the respective overall reactor body pressure drop in the operational design point. These values were compared with the overall reactor ΔP measured during actual operation, with solids, showing a satisfactory match both for AR and FR. The recorded pressure values along the empty reactor bodies were small due to the low design velocities (up to 2.4 m s^{-1}). The maximum values of pressure difference between the bottom of the reactors and their top was found to be in the order of magnitude of 1 mbar. This means that the kinetic pressure losses have little influence on the pressure measured along the reactor bodies during operation. Anyhow it needs to be kept in mind that the lower pressure transmitter is placed at a height of 14 cm from the reactor bottom; the bottom pressure is expected to be higher.

Another preliminary test campaign was finalized to check the reliability of the indirect solids flow/flux calculation realized by means of the pressure gradient measurements (Eq.1). The so determined solids flux was compared with the visual measurements of mass accumulation. It was of great interest for the project, in order to be capable to make use of this technique also in the 150kW_{th} hot rig, where it will not be so straightforward to have a visual measurement. The two measurement techniques matched when an auxiliary air injection below the perforated flap valve was tuned on purpose in order to achieve minimum fluidization conditions of the accumulated mass of particles. In addition, the agreement was mainly for the lower part of the solids flux range tested. In fact the momentum of the free falling solids was proven to affect strongly the fluidization of the accumulated powder in the downcomer [12]. A wide range of solids fluxes are going to be tested and they can't be foreseen in detail because of their dependency on many independent parameters. It means that we can't know in advance what the exact amount of auxiliary air will be. From these findings, and supported by literature [13], it was possible to deduct that this approach is very much depending on the operator ability of reaching the right fluidization conditions above the perforated flap valve. Thus it can't be used by itself for the hot rig and another solution applicable to high temperature conditions needs to be found.

It was noticed that it is difficult to obtain exactly the same solids flow/flux when the same experiments are not performed continuously; while they are very much consistent when executed continuously without changing settings and stopping the system. This fact shows that the "roughness" of the experimental technique used to measure the solids flow can not be the main reason for the variations in the results. The way the solids inventory distributes in the system in order to achieve steady state conditions do also play a role. A third issue affecting the solid flow is the Total Solids Inventory (TSI) control. It is closely related to the cyclones efficiency, evaluated to be very high and often above 99.9%. This is a good performance for such fine powder as can be seen in Fluid Catalytic Cracking (FCC) literature which is dealing with this kind of particle size distribution, but much lighter material [14]. Nevertheless it is worth to mention that for example in the case of a solid flow of 1 kg s^{-1} a cyclone efficiency of "just" 99.9% will mean 3.6 kg of losses in one hour. Such loss of mass will substantially affect the TSI and consequently the reactor performance. This information is of fundamental importance for the interpretation of the experimental results. The same set of tests was repeated twice for the AR with a TSI of 65kg and different refilling time of the lost mass. The results show that the 20 minutes refilling gave in average about 4% higher solids flow than the 60 minutes one (for a set of data of 20 points of which 8 are shown in Figure 2). Also, the consistent behaviour of the larger downcomer height confirms the indication that the solids flow difference is caused by an actual change in the reactor overall mass and mass distribution.

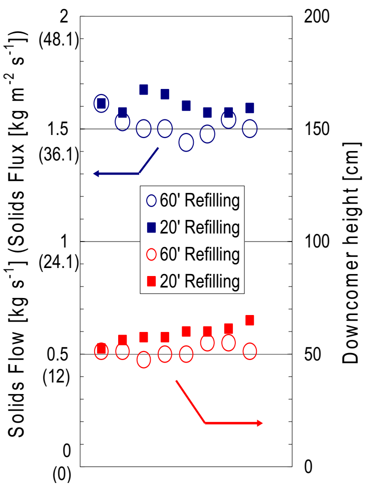

Figure 2. Set of tests performed twice with the same conditions, but with 60’ and 20’ refilling.

Next, the AR was run alone re-circulating internally through the divided loop-seal all the entrained solids. It was tested from part load, turbulent fluidization regime, up to an air flow corresponding to the fast CFB design flow regime. The solids flow/flux and the pressure profile along the reactor body were measured and the cyclone efficiency estimated. The TSI within the reactor system was varied as well as the combinations of primary and secondary air. The measured flow of solids was found to be clearly dependent on the TSI and the air flow, increasing with them up to 2 kg s^{-1} (flux of 48 kg m^{-2} s^{-1}). At the same time also the pressure behaviour showed its dependency from the TSI and air staging, shifting towards higher values for higher amount of solids and higher primary air share. In addition higher values of pressure gradient, thus mass, in the lower part of the reactor body are recorded, as expected [15], for turbulent regime, while the particle concentration in the upper part of the reactor increases when increasing the fluidization velocities. The same set of experiments was performed for the FR showing the same behaviour when it comes to solids entrainment and pressure behaviour. It was possible to entrain in a stable way a flow of particles up to almost half of the AR one, which is in accordance with the design. In average the bottom pressures of the two reactors running separately were shown to be on the same order of magnitude, slightly higher for the FR, picking the right operational conditions. This is an important parameter for the coupled operation control because this is the pressure where the return legs of the divided loop-seals merge into the reactor bodies. More details about the performance of the reactors operated separately can be found in Bischi et al. [2 and 9].

The way the loop-seal is operated affects quite a lot the performance of a CFB, and especially the solids entrainment [16 and 17]. For this reason the effect of the loop-seal fluidization was thoroughly investigated, varying the amount of fluidizing air injected in the central and internal nozzles for several lateral injections (that ones located in the downcomer just above the loop-seal). The aim was to understand the best way to operate the loop-seal for the actual reactor design. It means achieving high solids circulation with a stable fluidization regime in the downcomer (between minimum fluidization and minimum bubbling). For economical reasons this has to be done using a small amount of fluidizing gas because it will be steam in the hot rig. Figure 3 shows one detailed test matrix executed for a constant TSI of 55 kg in the AR operated separately (internal recirculation) and with a fluidizing air flow of 5000 Nl min^{-1}, equivalent to a superficial gas velocity of 2.2 m s^{-1}, and with an air split of 70% primary air, 15% secondary air 1 and 15% secondary air 2. The AR loop-seal will be the more heavily loaded according to the design. Each group of four points in the graph corresponds to a different value of the central nozzle fluidization (5 to 120 Nl min^{-1}) and for each of the groups the four points go from the value of 80 Nl min^{-1} up to 190 Nl min^{-1} of internal nozzle fluidization. Each different symbol (diamond, circle, and asterisk) refers to a different value of lateral air injection from 2.5 Nl min^{-1} up to 10 Nl min^{-1}. The blue points show (scale on the left) the measured solids flow/flux, while the red points show the measured height of solids in the downcomer. This is an important parameter to monitor in order to know how much of the mass is in the loop-seal downcomer rather than in the reactor body. It is possible to notice that the entrained flow of solids is appreciably increasing with the internal nozzle fluidization for the lower central fluidizations while above 40 Nl min^{-1} of central fluidization it stabilizes loosing its dependencies on the internal and central nozzles. For central nozzle fluidizations above 40 Nl min^{-1} it is not possible to distinguish clear trends, not even dependency from the lateral air injection amount. The downcomer height is consistently reduced with the increase of solids flow because, for a fixed TSI and reactor fluidization, a smoother loop-seal fluidization provides better solids circulation, thus more mass inside the reactor body, thus higher ΔP across the reactor and higher entrainment. On the other hand the higher pressure fluctuations experienced in the bottom of the loop-seal in correspondence with higher fluidizing air, e.g. central nozzle above 40 Nl min^{-1}, means that the reactor is exposed to higher risks of gas leakages and cyclone perturbations. A similar set of experiments was performed for the AR keeping the same air flows in the reactor and loop-seal, but increasing the TSI up to 65 kg. The increase of mass reduced the dependencies highlighted previously as well as increased the solids flow together with the amount of mass in the downcomer. The increase of the downcomer solids height is necessary in order to close the pressure loop; because more mass in the system gives a higher value of pressure in the reactor bottom exactly where the return leg is merging, thus higher pressure in the loop-seal is required to balance it. The loop-seal results are not following clear trends as in the cited literature [16 and 17] most likely because both the high density Geldart A particles and the fluidizing nozzles of the present set-up represent solutions differing from the majority of the published laboratory loop-seal studies. Therefore modifications of the loop-seal fluidizing system are under investigation in order to gain better control on the solids circulation, especially to tackle circumstances where the two solids streams exiting from the loop-seal are facing different pressures, as in the coupled operation.

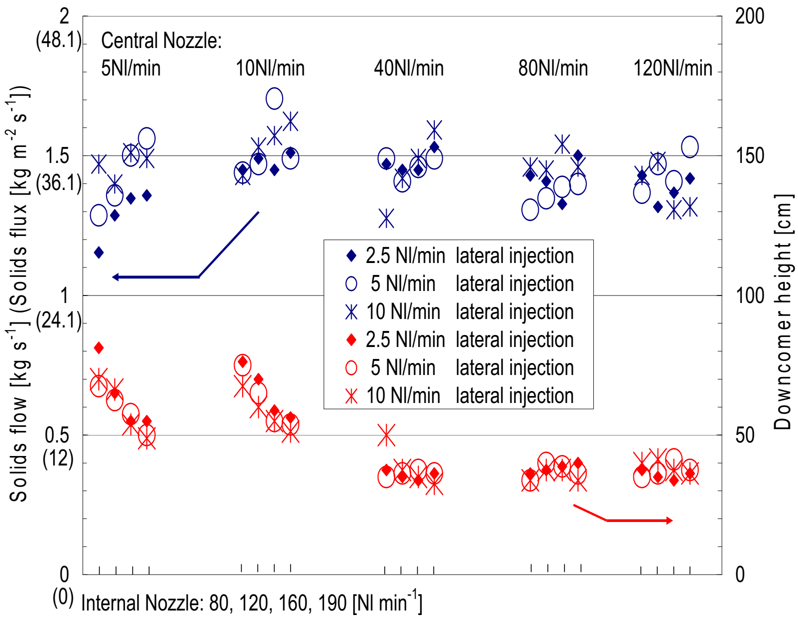

Figure 3. Set of experiments finalized to understand the air reactor dependency on the loop-seal operation (internal, central and lateral air injection) for a total solids inventory of 55 kg and air flow of 5000 Nl min^{-1}.

In this experimental campaign also the cooling panels influence on the AR performance was evaluated. Figure 4 shows the results of a test campaign conduced with a TSI of 65 kg, a total air flow going from 4000 to 5000 Nl min^{-1} and constant loop-seal fluidization. Tests were done with no cooling panels, with the lower (bott.), the middle (mid.) and the upper (up) panel separately and with the lower and middle together. The solids flow/flux was not significantly affected by panel insertion, location and number. The same applies for the measured average pressure values in the reactor body, while the pressure oscillations measured in the loop-seal bottom were in general higher for the two-panel configuration, e.g. above 20 mbar vs. 10 mbar. The test done with 100% of flow in the primary air (100%-0%-0%) showed a higher solids flow entrainment compared to the use of secondary air for the same amount of fluidizing air (50%-25%-25%). The use of only primary air was limited to 4000 Nl min^{-1} because further increase of flow generated pressure pulsations that made the system vibrating too much to operate it safely (test done just in the two panels case). It may be related to the inventory which needs to be reduced for such operational mode; further tests to proof it need to be carried out.

All the abovementioned tests were executed running the AR and FR separately. In this way it was possible to have an accurate mapping of their operational window and choose the best way to couple them together as a DLCFB reactor system. A test campaign with the two reactors coupled was performed but the results were not as expected. A high difference of pressure between the lower sections of the reactors was experienced: the FR bottom pressure ended up being much higher. It means that each divided loop-seal was exposed to a pressure unbalance having one return leg facing a pressure much higher than the other one. This fact sums up to the abovementioned loop-seal solids flux control challenges. The combination of these two circumstances created a disturbance because of gas flowing through the internal leg of the FR loop-seal, which is not in use during coupled operation with 100% solids exchange. It also generated a high pressure in the AR loop-seal external return leg, thus a high accumulation of particles in the AR downcomer capable to push the powder flow from the AR to the FR and very likely causing unwanted gas leakages from the FR to the AR. In addition it affected the cyclones efficiency causing mass losses and resulted in a loss of control of the system performance. An attempt to operate the system was done sealing the internal return legs of the loop-seals, without exposing them to the mentioned pressure unbalance. In this way the DLCFB reactor system reached automatically a stable configuration, showing good margins of operability. Afterwards the FR fluidizing system was modified, shifting the secondary air injections to a higher position. In this way the FR bottom pressure was reduced making the overall system more easily operable and the sealing of the internal return legs of the loops-seals could be removed. An example of the obtained pressure profiles are shown in Figure 5 and are between turbulent and fast CFB fluidization regimes. In the test shown in Figure 5 the TSI in the system was approximately 120kg while the mass inventories in the AR and FR were 19 kg and 15 kg, respectively. The mass in the reactor bodies was estimated by means of the measured pressure profiles, neglecting frictional and acceleration losses [18]. A solids flow of 1.65kg s^{-1} (corresponding to a flux of 40 kg m^{-2} s^{-1}) was entrained by the AR with a superficial gas velocity of 2.1 m s^{-1} while the FR entrained 0.85 kg s^{-1} (corresponding to a flux of 51 kg m^{-2} s^{-1}) with a superficial gas velocity of 2.2 m s^{-1}. The remaining 0.8 kg s^{-1} of particles flow necessary to achieve steady state were sent to the AR by means of the bottom lift/extraction operated with turbulent fluidization at 1 m s^{-1} of superficial gas velocity and a solids flux corresponding to 100 kg m^{-2} s^{-1}. More experiments to partially equilibrate

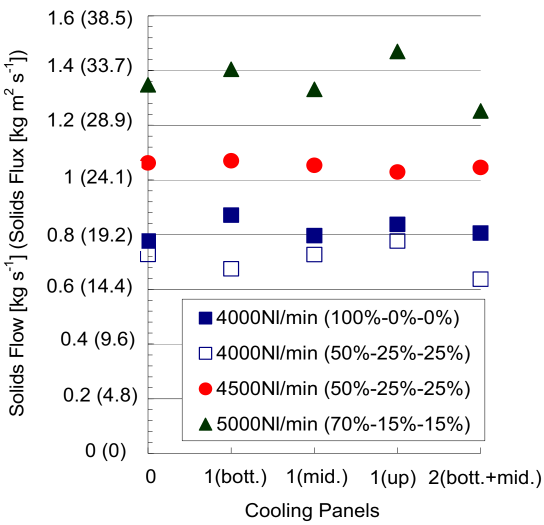

Figure 4. Solids flow/flux measurements with cooling panels insertions in different configurations.

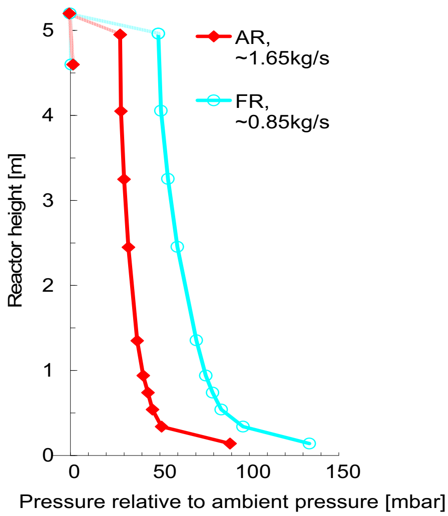

Figure 5. Pressure profiles measurement, coupled operation of Air Reactor (AR) and Fuel Reactor (FR).

the pressures between the lower sections of the reactors are on going. One of them is a reduction of the TSI which will decrease the pressures in the lower sections of the reactors. Another one is the utilization of the backpressure valves located at the cyclones exit in order to increase the AR backpressure, thus the pressure at the AR bottom. This solution will not be so straightforward because of all the interconnections between the AR and FR bodies, thus the pressure changes in one of them will affect to some extent also the other. Among the possible modifications of the loop-seals the introduction of a cone valve in each of their internal leg has proven to be an effective solution in order to face pressure difference between reactors. In fact the operation with a sealing can be considered equivalent to a cone valve fully closed. On the other hand, operating the DLCFB reactor system in a way which doesn't rely too much on active control (e.g. backpressure valves or cone valves) is more in line with the original design basis of the reactor system. Therefore the height where the loop-seals return legs, both the internal and external, are merging with the reactors can be lifted to a value where the pressure in the reactor bodies is decreased enough to make the system more easily operable with a wider stable operational window. This may cause residence time reduction and increase the risk of leakages of gas carried by the entrained solids from one reactor to the other [19], but will for sure increase the intrinsic stability of the system.

Conclusions and outlook

The full scale cold flow model of a second generation chemical looping combustion reactor system was commissioned and its performance with high density Geldart A particles was tested at a wide range of operating conditions.

The fluidizing system design was verified as well as the fraction of the kinetic losses on the overall reactor pressure drop. The suitability of an indirect measurement technique of the solids flow/flux entrainment was evaluated and compared to a more conventional direct one based on visual measurement of mass accumulation. A simplified error assessment of the direct solids flow/flux measurement was done, and the influence of the total solids inventory control and distribution on the measured values was highlighted. The cyclone efficiency was also estimated together with its influence on the abovementioned solids inventory control.

A comprehensive understanding of the stable operational window of the air and fuel reactor systems tested separately was obtained. The solids flow/flux entrainment and the pressure profiles along the air reactor and the fuel reactor were analyzed as well as their sensitivity to the parameters: superficial gas velocity, secondary air injection, solids inventory and loop-seal fluidization. Especially the way the loop-seal affects the reactors performance was systematically analyzed in order to find the best combination of air flow to the central, internal and lateral air injections. The loop-seals showed the capability of circulating the required solid flow, even if a clear trend was not found on how they ideally should be operated in order to attain a sharp and exact control. Therefore the fluidizing system of the loop-seals needs to be improved.

Furthermore, the overall double loop circulating fluidized bed reactor system performance was verified. A pressure difference was experienced between the lower sections of the two reactors, thus between the two loop-seals return legs. This made the operation of the overall system difficult. Better control was obtained sealing the internal legs of the divided loop-seals, as if a cone valve, 100% closed, was inserted. Finally, by modifying the FR secondary fluidization positions and changing the fluidizing air distribution it was possible to reduce the pressure unbalance and establish a stable solids exchange between the reactors. To that respect the system showed to be flexible and automatically adjusted the amount of solids in the downcomers to fulfil the overall pressure balance requirements.

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